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05/26/00 04:14 PM

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cc: Bill Maxwell/RTP/USEPA/US@EPA, jsnow@cinergy.com,  
PCHU@epri.com, metco@connect.net, bhuff@cinergy.com

Subject: Gibson Station Mercury Stack Test Report

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Dear Mr. Grimley,

As we discussed with you in March of this year, Cinergy elected to conduct a retest of the stack test on our Gibson Unit #3 in accordance with the Mercury ICR. As you recall, we found some unanticipated variability in results during our original stack test. We have since completed the retest and its report. Based on our analysis of the operating conditions and the test results, we have determined that the original test report is more suitable for characterizing mercury emissions from Gibson Station Unit #3 than the retest. We determined that certain operating conditions of a coal fired boiler can apparently greatly influence the amount and speciation of mercury that enters our precipitators. We are submitting to you the test reports for the March retest as well as the original test conducted in October of 1999.

This afternoon we sent three copies of each report to you via UPS next day delivery. The tracking number is: 1Z4921582210160496. These reports should arrive at your office on Tuesday, May 30th. Please call me if you have any questions.

J. Michael Geers  
Cinergy Environmental Services  
513.287.3839

Cinergy Corp.  
139 East Fourth Street  
P.O. Box 960  
Cincinnati, OH 45201-0960

May 26, 2000

Mr. William Grimley  
Emissions Measurement Center  
Interstate 40 and Page Road  
4930 Old Page Road  
Room Number E-108  
Durham, North Carolina 27709



Dear Mr. Maxwell,

Cinergy, as required in the Mercury Information Collection Request (ICR), has completed mercury speciation stack testing at Unit #3 of our Gibson Station. In March of this year, we submitted to you preliminary results from the test that we conducted in October of 1999. In summarizing our results to date, we identified significant variations in mercury concentration, particularly in the particulate fraction. While we had suspicions as to the cause, we could not directly attribute this variability to any changes in either the coal or the unit operation.

Because of our desire to provide accurate and representative information concerning Gibson #3 emissions, we elected to conduct a second stack test at the end of March. During that test we operated as close to the same conditions as the first test. However plant-operating conditions required that we test with air heater soot blowers operating. This did allow us to test one of our leading theories for the variability in this test. However after reviewing the data it apparently resulted in non-typical operating conditions.

We feel that taken as an average, the results of the October 1999 provide the most representative view of emissions from Gibson Unit #3. Under a separate cover we are also providing to you the results of the second stack test. We feel that the information, while collected under non-typical operating conditions, identifies issues concerning mercury emissions that require further investigate to completely understand mercury emissions from coal fired boilers. Please call me at 513-287-3839 or Paul Chu at 650-855-2812 if you would like to discuss.

Very truly yours,

A handwritten signature in dark ink, reading "J. Michael Geers, P.E." in a cursive style.

J. Michael Geers, P.E.  
Environmental Services Department

JMG

SOURCE EMISSIONS SURVEY  
OF  
GIBSON GENERATING STATION  
UNIT NUMBER 3 PRECIPITATOR INLET DUCT  
AND STACK  
OWENSVILLE, INDIANA  
FOR  
CINERGY CORPORATION  
AND  
THE ELECTRIC POWER RESEARCH INSTITUTE

OCTOBER 1999

FILE NUMBER 99-95GIB3

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## **1 INTRODUCTION**

### 1.1 Summary of Test Program

METCO Environmental, Dallas, Texas, conducted a source emissions survey of the Gibson Generating Station, Unit Number 3, located in Owensville, Indiana, for Cinergy Corporation and the Electric Power Research Institute, on October 13, 14, and 15, 1999. The purpose of these tests was to meet the requirements of the EPA Mercury Information Collection Request (ICR). Speciated mercury concentrations at the Unit Number 3 Precipitator Inlet Duct, speciated mercury emissions at the Unit Number 3 Stack, and mercury and chlorine content of the fuel were determined. The sulfur, ash, and Btu content of the fuel were also determined.

The sampling followed the procedures set forth in the Code of Federal Regulations, Title 40, Chapter I, Part 60, Appendix A, Methods 1, 2, 3B, 4, 5, 17, and 19; in the Ontario Hydro Method, Revised July 7, 1999; and ASTM Methods D2234, D6414-99, D2361-95, D-0516, D-3174, and D-3286. The test was also conducted in accordance with the Sampling and Analytical Test Plan and the Quality Assurance Project Plan previously submitted to, and approved by the US EPA.

### 1.2 Key personnel

Mr. Bill Hefley of METCO Environmental was the onsite project manager. Mr. Shane Lee, Mr. Mike Bass, Mr. Jason Conway, Mr. Scott Hart, and Mr. Jason Brown of METCO Environmental performed the testing.

Mr. J. Michael Geers of Cinergy Corporation was the utility representative. Mr. Jeffrey S. Snow of Cinergy Corporation performed process monitoring and sampling.

Mr. Paul Chu was the Electric Power Research Institute project manager.

**Table 1-1 Test Program Organization**

<b>Organization</b>	<b>Individual</b>	<b>Responsibility</b>	<b>Phone Number</b>
<i>Project Team</i> METCO	Bill Hefley	Project Manager	(972) 931-7127
<i>Utility</i> Cinergy Corp.	J. Michael Geers	Utility Representative	(513) 287-3839
Cinergy Corp.	Jeffrey S. Snow	Process Monitoring & Sampling	(812) 386-4202
<i>QA/QC</i> EPRI	Paul Chu	Project Manager	(650) 855-2812

## **2 SOURCE AND SAMPLING LOCATION DESCRIPTIONS**

### **2.1 Process Description**

Gibson Unit Number 3 is a Foster Wheeler opposed wall fired, supercritical, once through, balanced draft unit. The boiler is designed to operate at 4,588,000 pounds of steam per hour at 1,005 °F and 3,850 psig. Steam is supplied to a GE turbine-generator. The design gross capacity of the unit is 676 megawatts.

Gibson Generating Station receives Coal from train and truck deliveries. Coal is also stored on-site to provide a reliable supply during delivery disruptions. Coal is conveyed via conveyor belts to a coal storage bunker inside the plant. During the conveyance to the bunker, the coal is sampled by a mechanical sampling system. From the bunkers, the coal is fed into the pulverizers by variable speed belt feeders that control coal flow rate. The pulverizers crush the coal the fineness necessary for combustion in the boiler.

Combustion air is drawn from the atmosphere by forced draft fans. At the pulverizers, a portion of the combustion air is used to transport the finely crushed coal to the burners in the boiler. The remainder of the air is introduced directly at the boiler. The heat released during combustion is used to create steam that flows to a turbine-generator to produce electricity.

After combustion, the gasses generated (flue gas) are drawn from the boiler by induced draft fans. The combination of forced and induced draft fans is balanced to produce a neutral gas pressure in the boiler. As the flue gas exits the boiler, an air heater removes a portion of the waste heat. The air heater transfers the captured waste heat to the incoming combustion air.



## 2.2 Control Equipment Description

Flue gas flows into the electrostatic precipitators to remove particulate matter. The electrostatic precipitator (ESP) electrically charges the ash particles in the flue gas to collect and remove them. The unit is comprised of a series of parallel vertical plates through which the flue gas passes. Centered between the plates are charging electrodes which provide the electric field. As flue gas passes through the electric field, the particulate material takes on a negative charge. The ash particles are then attracted to the grounded collection plates. The particles form a layer of ash on the collection plates. Periodically the collection plates are mechanically rapped causing the accumulated ash to fall into collection hoppers below. The collected ash is then evacuated from the hopper by the fly ash transport system.

## 2.3 Flue Gas and Process Sampling Locations

### *2.3.1 Inlet Sampling Locations*

The sampling location on the Unit Number 3 Precipitator Inlet Duct is approximately 83 feet above the ground. The sampling location is located 24 feet 11 inches (1.88 equivalent duct diameters) downstream from a bend in the duct and 21 feet 7 inches (1.63 equivalent duct diameters) upstream from a bend in the duct.

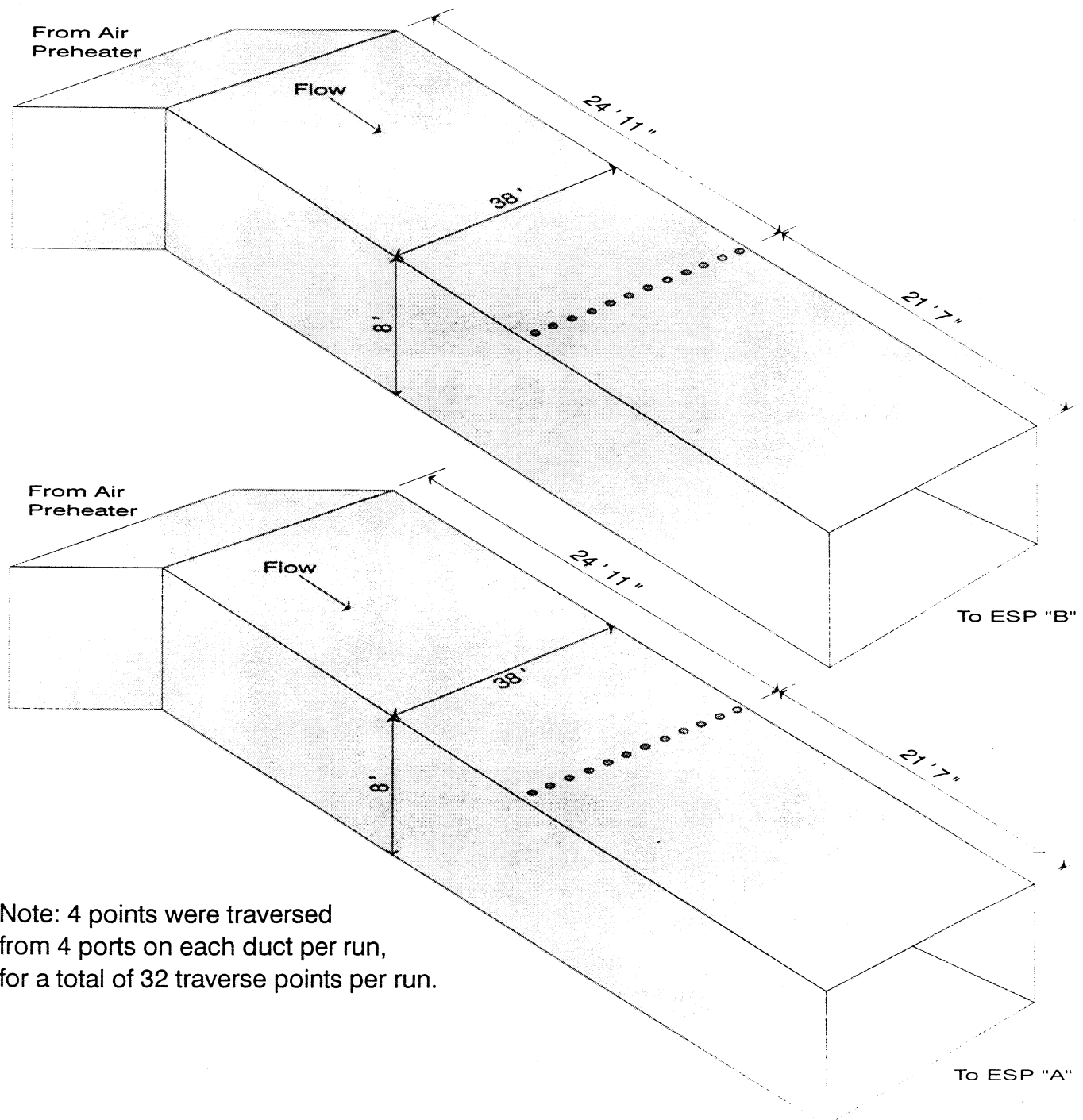
### *2.3.2 Stack Sampling Locations*

The sampling location on the Unit Number 3 Stack is approximately 252 feet 6 ½ inches above the ground. The sampling locations are located 155 feet 8 inches (4.88 stack diameters) downstream from inlet to the stack and 252 feet 5 inches (7.91 stack diameters) upstream from the outlet to the stack.

### *2.3.3 Coal Sampling Location*

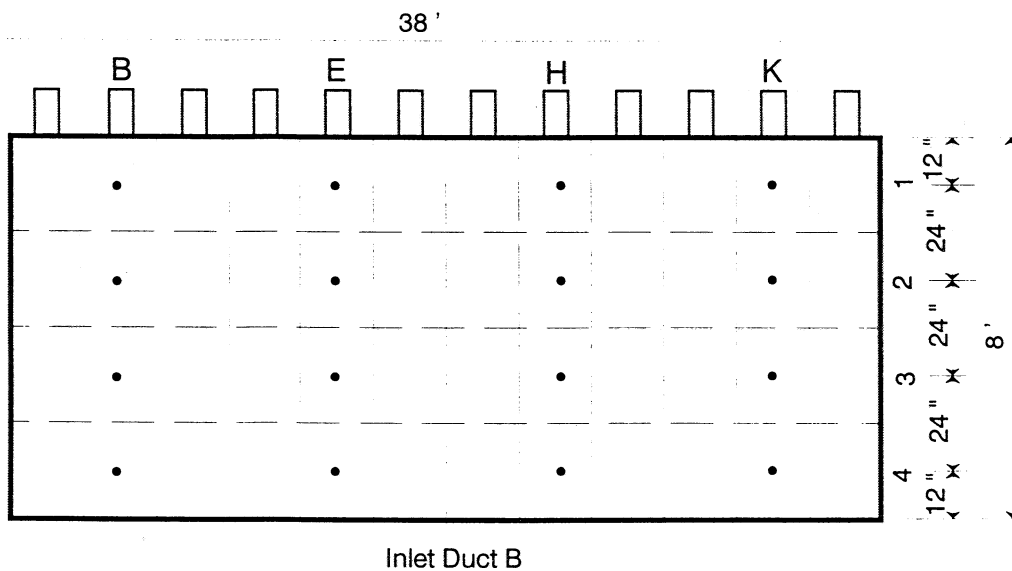
The coal sampling locations are located at the coal feeders and immediately downstream of the coal bunkers (B).

**Figure 2-1**  
**Description of sampling locations at Gibson Unit Number 3 Precipitator Inlet Duct**

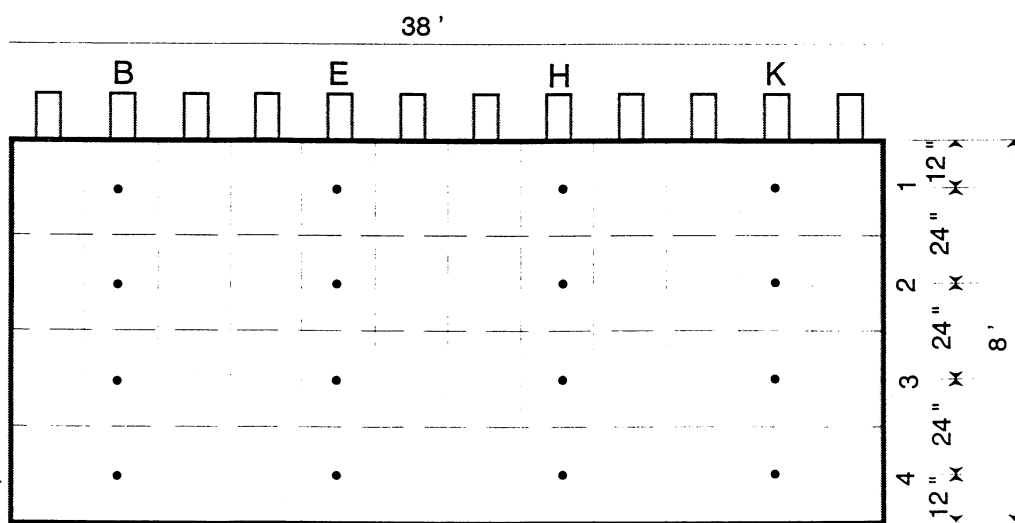


Note: 4 points were traversed  
 from 4 ports on each duct per run,  
 for a total of 32 traverse points per run.

**Figure 2-2**  
**Description of sampling points at Gibson Unit Number 3 Precipitator Inlet Duct**

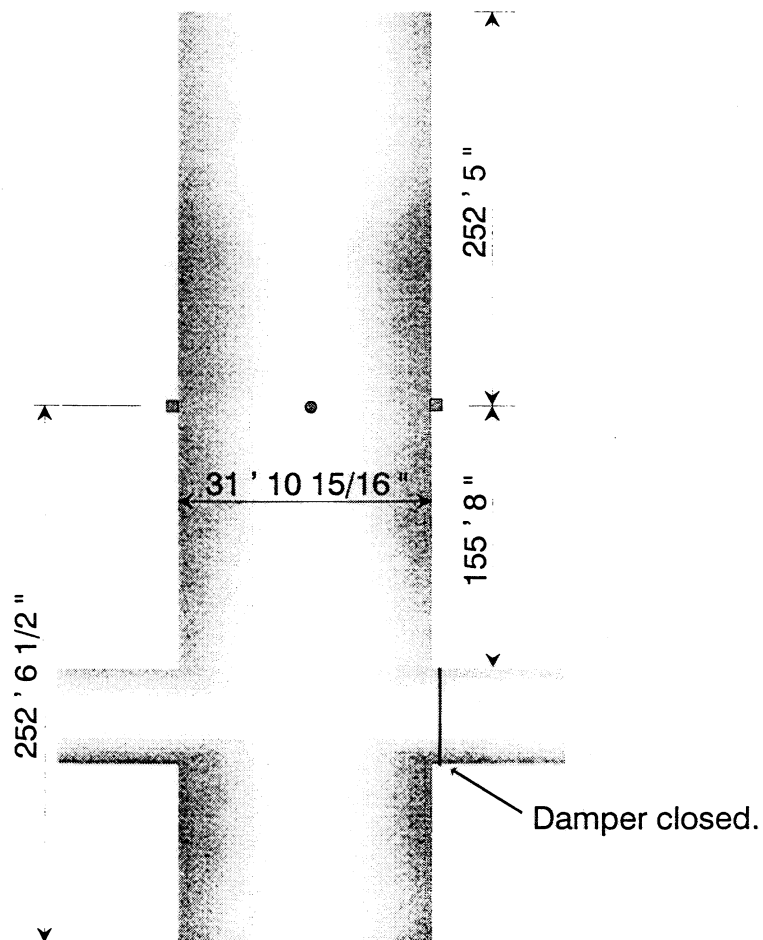


Inlet Duct B

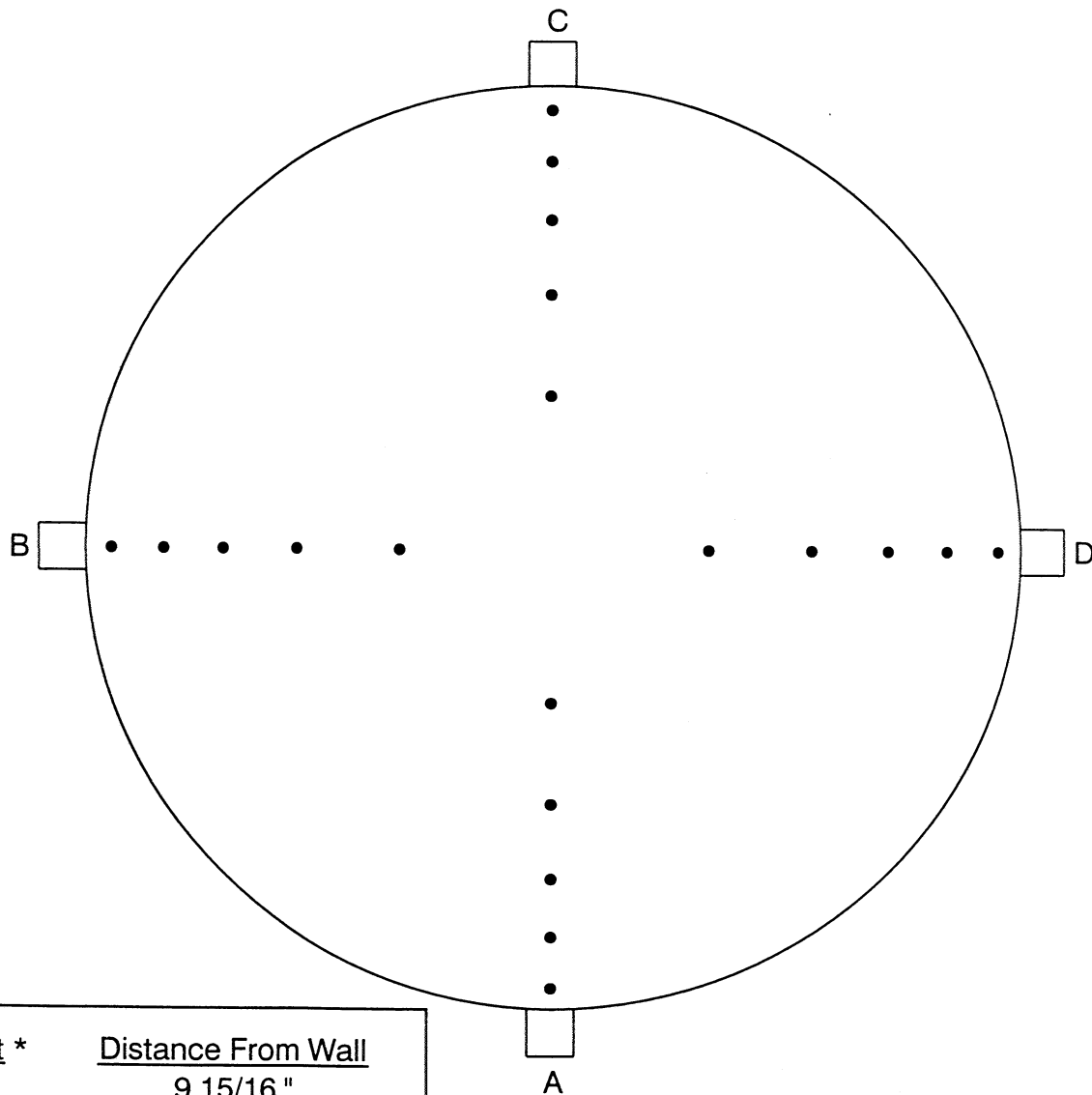


Inlet Duct A

**Figure 2-3**  
**Description of sampling locations at Gibson Unit Number 3 Stack**



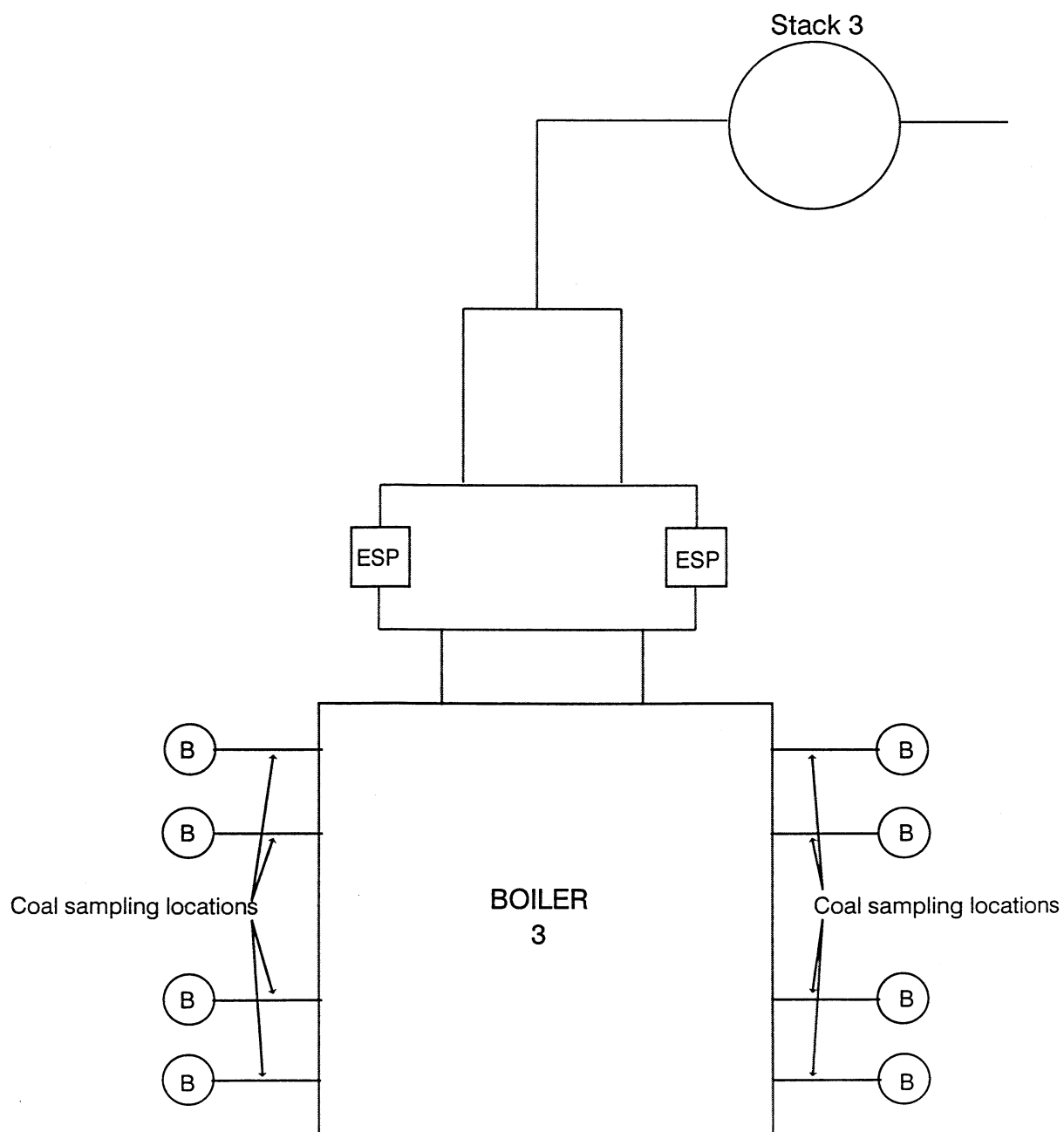
**Figure 2-4**  
**Description of sampling points at Gibson Unit Number 3 Stack**



<u>Point *</u>	<u>Distance From Wall</u>
1	9 15/16 "
2	31 3/8 "
3	55 15/16 "
4	86 9/16 "
5	130 15/16 "

\* Calculated as one-half of a ten point traverse.

**Figure 2-5**  
**Description of coal sampling locations at Gibson Unit Number 3**



### **3 SUMMARY AND DISCUSSION OF RESULTS**

#### **3.1 Objectives and Test Matrix**

##### ***3.1.1 Objective***

The objective of the tests was to collect the information and measurements required by the EPA Mercury ICR. Specific objectives listed in order of priority are:

1. Quantify speciated mercury emissions at the stack.
2. Quantify speciated mercury concentrations in the flue gas at the inlet.
3. Quantify fuel mercury and chlorine content during the stack and inlet tests.
4. Provide the above information for use in developing boiler, fuel, and specific control device mercury emission factors.

##### ***3.1.2 Test Matrix***

The test matrix is presented in Table 1. The table includes a list of test methods to be used. In addition to speciated mercury, the flue gas measurements include moisture, flue gas flow rates, carbon dioxide, and oxygen.



**Table 3-1**  
**Test Matrix for Mercury ICR Tests at Gibson Unit Number 3**

Sampling Location	No. of Runs	Species Measured	Sampling Method	Sample Run Time	Analytical Method	Analytical Laboratory
Stack	3	Speciated Hg	Ontario Hydro	160 min	Ontario Hydro	TestAmerica
Stack	3	Moisture	EPA 4	Concurrent	Gravimetric	METCO
Stack	3	Flue Gas Flow	EPA 1 & 2	Concurrent	Pitot Traverse	METCO
Stack	3	O <sub>2</sub> & CO <sub>2</sub>	EPA 3B	Concurrent	Orsat	METCO
Inlet	3	Speciated Hg	Ontario Hydro	160 min	Ontario Hydro	TestAmerica
Inlet	3	Moisture	EPA 4	Concurrent	Gravimetric	METCO
Inlet	3	Flue Gas Flow	EPA 1 & 2	Concurrent	Pitot Traverse	METCO
Inlet	3	O <sub>2</sub> & CO <sub>2</sub>	EPA 3B	Concurrent	Orsat	METCO
Coal Feeders	3	Hg, Cl, Sulfur, Ash, and Btu/lb in coal	ASTM D2234	1 grab sample every 30-minutes per feeder per run	ASTM D6414-99 (Hg), ASTM D2361-95 (Cl), ASTM D-0516 (S), ASTM D-3174 (Ash), and ASTM D-3286 (Btu/lb)	TestAmerica and Philip Services

### 3.2 Field Test Changes and Problems

Run Number 2 was aborted due to reference method sampling equipment problems.

### 3.3 Handling of Non-Detects

This section addresses how data will be handled in cases where no mercury is detected in an analytical fraction. It should be noted that the analytical method specified in the Ontario Hydro Method has a very low detection limit, which is expected to be well below flue gas levels for most cases if the laboratory uses normal care and state of the art analytical equipment. However, there were cases where certain fractions of a test did not show detectable mercury levels. This section addresses how non-detects were handled in calculating and reporting mercury levels.

#### *3.3.1 A single analytical fraction representing a subset of a mercury species is not detected.*

When more than one sample component is analyzed to determine a mercury species (such as analyzing the probe rinse and filter catch separately to determine total particulate mercury) and one fraction is not detected, it will be counted as zero. Total mercury for that species will be the sum of the detected values of the remaining fraction(s). For example, if the probe rinse had ND < 0.05 µg and the filter had 1.5 µg, total particulate mercury would be reported as 1.5 micrograms.

#### *3.3.2 All fractions representing a mercury species are not detected.*

If all fractions used to determine a mercury species are not detected, the total mercury for that species will be reported as not detected, at the sum of the detection limits of the individual species.

For example, if the probe rinse were not detected at 0.003  $\mu\text{g}$  and the filter catch were not detected at 0.004  $\mu\text{g}$ , the reported particulate mercury would be reported as ND < 0.007  $\mu\text{g}$ . This is expected to represent a small fraction (<1%) of the total mercury, even under worse case scenario of 1  $\mu\text{g}/\text{Nm}^3$ .

### *3.3.3 No mercury is detected for a species on all three test runs.*

When all three test runs show no detectable levels of mercury for a mercury species, that mercury species will be reported as not detected at less than the average detection limit. For example, if three results for elemental mercury are ND < 0.10, ND < 0.13, and ND < 0.10, the results would be reported as ND < 0.13 (the highest of the three detection levels).

In calculating total mercury, a value of zero will be used for that species. For example, if particulate mercury were ND < 0.11  $\mu\text{g}$ , oxidized mercury were 2.0  $\mu\text{g}$ , and elemental mercury were 3.0  $\mu\text{g}$ , total mercury would be reported as 5.0  $\mu\text{g}$ .

In calculating the percentage of mercury in the other two species, a value of zero will be used. For the example listed in the preceding paragraph, the results would be reported as 0% particulate mercury, 40% oxidized mercury, and 60% elemental mercury.

### *3.3.4 Mercury is detected on one or two of three runs.*

If mercury is detected on one or two of three runs, average mercury will be calculated as the average of the detected value(s) and half of the detection limits for the non-detect(s).

Example 1: The results for three runs are 0.20, 0.20, and ND < 0.10. The reported value would be calculated as the average of 0.20, 0.20, and 0.05, which is 0.15  $\mu\text{g}$ .

Example 2: The results for three runs are 0.14, ND < 0.1, and ND < 0.1. The average of 0.14, 0.05, and 0.05 is calculated to be 0.08. Since this is below the detection limit of 0.1, the reported value is ND < 0.1.

### 3.4 Summary of Results

The results of the tests performed at Gibson Unit Number 3 are listed in the following tables.

The 0.1 N nitric acid rinses (Containers Number 2) and the acetone rinses (Container Number 2A) were not combined prior to analysis for Run Numbers 1, 3, and 4 for the inlet and Run Number 1 for the stack. During separate analysis, the front half rinse samples were lost in the preparation step.

Due to insufficient particulate matter on the thimble samples from the Unit Number 3 Stack, the entire thimbles were digested, opposed to an aliquot as outlined in the Ontario Hydro Method. The large thimbles absorbed all of the acid used in digestion and overheated in the microwave rupturing the microwave membrane. The thimbles and the acid were recovered and additional acid was added. The samples were redigested. Although the digestion was done within hold time, the analysis was completed two days past hold time. Quartz fiber thimbles were used as a probe extension due to the required distance to the sample points on the Unit Number 3 Stack. For these reasons, the results of these fractions should be viewed as estimated since the amount of mercury lost, if any was not known.

The two thimble filters provided for the Unit Number 3 Precipitator Inlet Duct Run Number 1 were not composited prior to analysis. The samples were analyzed separately and mathematically combined.

**Table 3-2**  
**Gibson Unit Number 3 Source Emissions Results**

Run Number	1	3	4
Test Date	10/14/99	10/15/99	10/15/99
Test Time	0833-1303	0900-1247	1415-1736
<b>Inlet Gas Properties</b>			
Flow Rate - ACFM	2,303,170	2,265,550	2,378,770
Flow Rate - DSCFM*	1,379,200	1,385,045	1,395,885
% Water Vapor - % Vol.	7.92	4.63	8.20
CO <sub>2</sub> - %	13.6	14.0	14.0
O <sub>2</sub> - %	5.6	5.4	5.2
% Excess Air @ Sampling Point	35	34	32
Temperature - °F	317	323	326
Pressure - "Hg	28.53	28.34	28.37
Percent Isokinetic	102.8	97.7	98.5
Volume Dry Gas Sampled - DSCF*	81.137	59.274	59.540
<b>Stack Gas Properties</b>			
Flow Rate - ACFM	2,510,265	2,848,403	3,462,778
Flow Rate - DSCFM*	1,523,212	1,738,077	2,065,309
% Water Vapor - % Vol.	6.60	5.69	7.26
CO <sub>2</sub> - %	13.2	13.2	13.4
O <sub>2</sub> - %	5.9	6.0	5.8
% Excess Air @ Sampling Point	38	39	37
Temperature - °F	340	339	344
Pressure - "Hg	29.34	29.19	29.19
Percent Isokinetic	94.3	100.0	95.1
Volume Dry Gas Sampled - DSCF*	86.232	75.586	85.384

\* 29.92 "Hg, 68 °F (760 mm Hg, 20 °C)

Note: Run Number 2 was aborted due to reference method sampling equipment problems.

**Table 3-3**  
**Gibson Unit Number 3 Mercury Removal Efficiency**

Run Number	1	3	4	Average
Test Date	10/14/99	10/15/99	10/15/99	
Test Time	0833-1303	0900-1247	1415-1736	
<b>Total mercury</b>				
Inlet - lb/10 <sup>12</sup> Btu	12.99	23.27	12.27	16.18
Stack - lb/10 <sup>12</sup> Btu	7.91	9.58	11.19	9.56
Removal efficiency - %	39.1	58.8	8.8	35.6
<b>Particulate mercury</b>				
Inlet - lb/10 <sup>12</sup> Btu	3.95	19.67	3.28	8.97
Stack - lb/10 <sup>12</sup> Btu	<0.04	<0.07	<0.04	<0.07
Removal efficiency - %	>99.0	>99.6	>98.8	>99.2
<b>Oxidized mercury</b>				
Inlet - lb/10 <sup>12</sup> Btu	7.37	2.70	7.86	5.98
Stack - lb/10 <sup>12</sup> Btu	4.32	6.00	7.87	6.06
Removal efficiency - %	41.4	-----	-----	-----
<b>Elemental mercury</b>				
Inlet - lb/10 <sup>12</sup> Btu	1.67	0.89	1.13	1.23
Stack - lb/10 <sup>12</sup> Btu	3.59	3.57	3.32	3.49
Removal efficiency - %	-----	-----	-----	-----

**Table 3-4**  
**Gibson Unit Number 3 Mercury Speciation Results**

Run Number	1	3	4	Average
Test Date	10/14/99	10/15/99	10/15/99	
Test Time	0833-1303	0900-1247	1415-1736	
<b>Inlet Mercury Speciation</b>				
Particulate mercury – µg	10.87	40.10	6.81	—
µg/dscm	4.73	23.89	4.04	10.89
lb/10 <sup>12</sup> Btu	3.95	19.67	3.28	8.97
% of total Hg	30.4	84.5	26.7	47.2
Oxidized mercury – µg	20.30	5.51	16.30	—
µg/dscm	8.84	3.28	9.67	7.26
lb/10 <sup>12</sup> Btu	7.37	2.70	7.86	5.98
% of total Hg	56.7	11.6	64.1	44.2
Elemental mercury – µg	4.60	1.82	2.34	—
µg/dscm	2.00	1.08	1.39	1.49
lb/10 <sup>12</sup> Btu	1.67	0.89	1.13	1.23
% of total Hg	12.9	3.8	9.2	8.6
Total mercury – µg	35.77	47.43	25.45	—
µg/dscm	15.57	28.26	15.09	19.64
lb/10 <sup>12</sup> Btu	12.99	23.27	12.27	16.18
<b>Stack Mercury Speciation</b>				
Particulate mercury – µg	<0.11	<0.18	<0.11	—
µg/dscm	<0.05	<0.08	<0.05	<0.08
lb/10 <sup>12</sup> Btu	<0.04	<0.07	<0.04	<0.07
% of total Hg	<0.5	<0.7	<0.4	<0.5
Oxidized mercury – µg	12.40	15.00	22.50	—
µg/dscm	5.08	7.01	9.31	7.13
lb/10 <sup>12</sup> Btu	4.32	6.00	7.87	6.06
% of total Hg	54.6	62.6	70.3	62.5
Elemental mercury – µg	10.30	8.93	9.51	—
µg/dscm	4.22	4.17	3.93	4.11
lb/10 <sup>12</sup> Btu	3.59	3.57	3.32	3.49
% of total Hg	45.4	37.3	29.7	37.5
Total mercury – µg	22.70	23.93	32.01	—
µg/dscm	9.30	11.18	13.24	11.24
lb/10 <sup>12</sup> Btu	7.91	9.58	11.19	9.56
<b>Coal Analysis</b>				
Mercury – ppm dry	0.134	0.142	0.141	0.139
Mercury - lb/10 <sup>12</sup> Btu	12.10	13.33	13.42	12.95
Chlorine - ppm dry	1,900	2,200	2,200	2,100
Moisture - %	13.6	12.6	12.8	13.0
Sulfur - % dry	1.71	1.74	1.71	1.72
Ash - % dry	13.1	12.9	13.1	13.0
HHV - Btu/lb as fired	10,820	10,980	10,960	10,920
Coal flow - lb/hr as fired	534,600	546,600	544,000	541,733
Total Heat Input – 10 <sup>6</sup> Btu/hr	5,784.4	6,001.7	5,962.2	5,916.1
<b>Total Mercury Mass Rates</b>				
lb/hr input in coal	0.07	0.08	0.08	0.08
lb/hr at Precipitator inlet	0.08	0.15	0.08	0.10
lb/hr emitted	0.05	0.07	0.10	0.08

Note: Values reported as less than values represent detection limits.

**Table 3-5  
Gibson Unit Number 3 Process Data**

Run Number	1	3	4
Test Date	10/14/99	10/15/99	10/15/99
Test Time	0833-1303	0900-1247	1415-1736
<b>Unit Operation</b>			
Unit Load - MW net	640	641	639
Coal Mills in Service	5	5	5
Coal Flow - tons/hr	267.3	273.3	272.0
Steam Flow - klb/hr	4,428.1	4,398.8	4,394.1
Furnace Exit Gas Temp. - °F	789.8	793.1	791.7
<b>CEM data</b>			
CO <sub>2</sub> - % wet	12.3	12.3	12.3
SO <sub>2</sub> - lbs/10 <sup>12</sup> Btu	2.73	2.70	2.70
NO <sub>x</sub> - lbs/10 <sup>12</sup> Btu	0.49	0.46	0.45
Opacity - %	14.9	13.6	13.9
Flow - scf/hr	98,149,313	97,123,514	97,658,897
<b>Precipitator data</b>			
Opacity - %	12.8	11.3	11.3
Gas Inlet Temp. A - °F	313	312	320
Gas Inlet Temp. B - °F	335	334	341
Gas Outlet Temp. - °F	312	311	321



## **4 SAMPLING AND ANALYTICAL PROCEDURES**

### **4.1 Emission Test Methods**

The sampling followed the procedures set forth in the Code of Federal Regulations, Title 40, Chapter I, Part 60, Appendix A, Methods 1, 2, 3B, 4, 5, 17, and 19; in the Ontario Hydro Method, Revised July 7, 1999; and ASTM Methods D2234, D6414-99, D2361-95, D-0516, D-3174, and D-3286.

A preliminary velocity traverse was made at eight ports at the inlet sampling location, in order to determine the uniformity and magnitude of the flow prior to testing. All traverse points were checked for cyclonic flow and the average angle was equal to 1.0 degrees. Alternate procedures would be required if the angle of cyclonic flow was greater than 20 degrees. Four traverse points were sampled from each of the eight ports, for a total of thirty-two traverse points at both sampling locations.

A preliminary velocity traverse was made at each of the four ports at the stack sampling locations, in order to determine the uniformity and magnitude of the flow prior to testing. All traverse points were checked for cyclonic flow and the average angle was equal to 0.8 degrees. Alternate procedures would be required if the angle of cyclonic flow was greater than 20 degrees. Five traverse points were sampled from each of the four ports, for a total of twenty traverse points.

The sampling trains were leak-checked at the end of the nozzle at 15 inches of mercury vacuum before each test, and again after each test at the highest vacuum reading recorded during each test. This was done to predetermine the possibility of a diluted sample.

The pitot tube lines were checked for leaks before and after each test under both a vacuum and a pressure. The lines were also checked for clearance and the manometer was zeroed before each test.

Integrated orsat samples were collected and analyzed according to EPA Method 3B during each test.

#### *4.1.1 Mercury*

Triplicate samples for mercury were collected. The samples were taken according to EPA Methods 1, 2, 3B, 4, 5, and 17; and the Ontario Hydro Method, Revised July 7, 1999. For each run at the inlet sampling location, samples of five-minute duration were taken isokinetically at each of the thirty-two traverse points for a total sampling time of 160 minutes. For each run at the stack sampling location, samples of eight-minute duration were taken at each of the twenty traverse points for a total sampling time of 160 minutes. Data was recorded at four-minute intervals. Blank train samples and reagent blanks were submitted.

The "front-half" of the sampling train at the inlet sampling location contained the following components:

- Teflon Coated Nozzle
- In-stack Quartz Fiber Thimble and Backup Filter and Teflon Coated Support
- Heated Glass Probe @ > 248°F

The "front-half" of the sampling train at the outlet sampling location contained the following components:

- Teflon Coated Nozzle
- In-stack Quartz Fiber Thimble and Backup Filter and Teflon Coated Support
- Heated Glass Probe @ > 248°F

The "back-half" of the sampling train contained the following components:

<u>Impinger Number</u>	<u>Impinger Type</u>	<u>Impinger Contents</u>	<u>Amount</u>	<u>Parameter Collected</u>
1	Modified Design	1 mol/L KCL	100 ml	Oxidized Mercury and Moisture
2	Modified Design	1 mol/L KCL	100 ml	Oxidized Mercury and Moisture
3	Greenburg-Smith Design	1 mol/L KCL	100 ml	Oxidized Mercury and Moisture
4	Modified Design	5% HNO <sub>3</sub> and 10% H <sub>2</sub> O <sub>2</sub>	100 ml	Elemental Mercury and Moisture
5	Modified Design	4% KMnO <sub>4</sub> and 10% H <sub>2</sub> SO <sub>4</sub>	100 ml	Elemental Mercury and Moisture
6	Modified Design	4% KMnO <sub>4</sub> and 10% H <sub>2</sub> SO <sub>4</sub>	100 ml	Elemental Mercury and Moisture
7	Greenburg-Smith Design	4% KMnO <sub>4</sub> and 10% H <sub>2</sub> SO <sub>4</sub>	100 ml	Elemental Mercury and Moisture
8	Modified Design	Silica	200 g	Moisture

All glassware was cleaned prior to use according to the guidelines outlined in EPA Method 29, Section 5.1.1 and the Ontario Hydro Method, Revised July 7, 1999, Section 13.2.15. All glassware connections were sealed with Teflon tape.

At the conclusion of each test, the filter and impinger contents were recovered according to procedures outlined in the Ontario Hydro Method, Revised July 7, 1999, Section 13.2.

Mercury samples were analyzed by Cold Vapor Atomic Absorption and Fluorescence Spectroscopy.

#### 4.2 Process Test Methods

ASTM D2234 method of coal sampling was followed. For each test run, a grab sample of coal was collected every 30 minutes from each coal scale immediately downstream of the coal bunkers. One composite sample was prepared for analysis from the individual feeder samples. Each sample was analyzed for mercury, chlorine, sulfur, ash, and Btu content by ASTM Methods D6414-99, D2361-95, D-0516, D-3174, and D-3286, respectively.

#### 4.3 Sample Tracking and Custody

Samples and reagents were maintained in limited access, locked storage at all times prior to the test dates. While on site, they were at an attended location or in an area with limited access. Off site, METCO and TestAmerica provided limited access, locked storage areas for maintaining custody.

Chain of custody forms are located in Appendix F. The chain of custody forms provide a detailed record of custody during sampling, with the initials noted of the individuals who loaded and recovered impinger contents and filters, and performed probe rinses.

## 5 QA/QC ACTIVITIES

The major project quality control checks are listed in Table 5-1. Matrix Spike Summaries are listed in Table 5-2. Duplicate and Triplicate Analyses Summaries are listed in Table 5-3. Additional method-specific QC checks are presented in Table 5-4 (Methods 1 and 2), Table 5-5 (Method 5/17 sampling), and Table 5-6 (Ontario Hydro sample recovery and analysis). These tables also include calibration frequency and specifications.

**Table 5-1**  
**Major Project Quality Control Checks**

<i><b>QC Check</b></i>	<i><b>Information Provided</b></i>	<i><b>Results</b></i>
<i><b>Blanks</b></i>		
Reagent blank	Bias from contaminated reagent	No Mercury was detected
Field blank	Bias from handling and glassware	No Mercury was detected
<i><b>Spikes</b></i>		
Matrix spike	Analytical bias	Sample results were between 75% - 125% recovery
<i><b>Replicates</b></i>		
Duplicate analyses	Analytical precision	Results were < 10% RPD
Triplicate analyses	Analytical precision	Results were < 10% RPD

**Table 5-2**  
**Gibson Unit Number 3 Matrix Spike Summary**

<i>Sampling Location</i>	<i>Run Number</i>	<i>Container</i>	<i>Results (<math>\mu</math>g)</i>	<i>True Value (<math>\mu</math>g)</i>	<i>Recover (%)</i>
Inlet Duct	1	2	0.927	0.900	103
Stack	1	3	6.41	7.45	86
Stack	1	4	3.15	3.00	105
Inlet Duct	4	1A	5.50	5.67	97
Inlet Duct	1	1B	0.0525	0.05	105
Stack	3	1A	0.058	0.05	116

**Table 5-3**  
**Gibson Unit Number 3 Duplicate and Triplicate Analyses Summary**

<i>Sampling Location</i>	<i>Run Number</i>	<i>Container</i>	<i>Results (µg)</i>	<i>Duplicate Results (µg)</i>	<i>RPD</i>	<i>Triplicate Results (µg)</i>	<i>RPD</i>
Inlet	1	1A	1.18	1.20	1.4	---	---
		1A	9.65	9.64	0.1	9.64	0.1
		1B	0.04	0.04	0.8	---	---
		2	<0.180	<0.180	0	---	---
		3	20.3	20.1	1.1	---	---
		4	<0.68	<0.68	0	---	---
		5	4.60	4.55	1	---	---
	3	1A	40.1	40.5	0.9	---	---
		1B	<0.01	<0.01	0	---	---
		2	<0.104	<0.104	0	---	---
		3	5.51	5.66	2.8	---	---
		4	<0.76	<0.76	0	---	---
		5	1.82	1.84	1.4	---	---
	4	1A	6.81	6.81	0	---	---
		1B	<0.01	<0.01	0	---	---
		2	<0.162	<0.162	0	---	---
		3	16.3	16.2	0.4	16.1	1.3
		4	<0.78	<0.78	0	---	---
		5	2.34	2.36	0	---	---
Stack	1	1A	<0.01	<0.01	0	<0.01	0
		1B	<0.01	<0.01	0	---	---
		2	<0.094	<0.094	0	---	---
		3	12.40	12.14	2.4	---	---
		4	<0.60	<0.60	0	---	---
		5	10.3	10.3	0	---	---
	3	1A	<0.01	<0.01	0	---	---
		1B	<0.01	<0.01	0	---	---
		2	<0.164	<0.164	0	---	---
		3	15.0	14.9	1.1	---	---
		4	<0.68	<0.68	0	---	---
		5	8.93	9.03	1.1	---	---
	4	1A	<0.01	<0.01	0	---	---
		1B	<0.01	<0.01	0	---	---
		2	<0.094	<0.094	0	---	---
		3	22.5	22.4	0.3	---	---
		4	<0.72	<0.72	0	---	---
		5	9.51	9.36	1.6	---	---

**Table 5-4**  
**QC Checklist and Limits for Methods 1 and 2**

Quality Control Activity	Acceptance Criteria and Frequency	Reference
Measurement site evaluation	>2 diameters downstream and 0.5 diameters upstream of disturbances*	Method 1, Section 2.1
Pitot tube inspection	Inspect each use for damage, once per program for design tolerances	Method 2, Figures 2-2 and 2-3
Thermocouple	+/- 1.5% (°R) of ASTM thermometer, before and after each test mobilization	Method 2, Section 4.3
Barometer	Calibrate each program vs. mercury barometer or vs. weather station with altitude correction	Method 2, Section 4.4

\* Although the inlet sampling location does not meet the requirements of EPA Method 1, three-dimensional flow testing as described in EPA Method 1 was not performed. All traverse points were checked for cyclonic flow and the average angle was equal to 1.0 degrees.



**Table 5-5**  
**QC Checklist and Limits for Method 5/17 Sampling**

Quality Control Activity	Acceptance Criteria and Frequency	Reference
<i>Pre-mobilization checks</i>		
Gas meter/orifice check	Before test series, $Y_D \pm 5\%$ (of original $Y_D$ )	Method 5, Section 5.3
Probe heating system	Continuity and resistance check on element	
Nozzles	Note number, size, material	
Glassware	Inspect for cleanliness, compatibility	
Thermocouples	Same as Method 2	
<i>On-site pre-test checks</i>		
Nozzle	Measure inner diameter before first run	Method 5, Section 5.1
Probe heater	Confirm ability to reach temperature	
Pitot tube leak check	No leakage	Method 2, Section 3.1
Visible inspection of train	Confirm cleanliness, proper assembly	
Sample train leak check	$\leq 0.02$ cf at 15" Hg vacuum	Method 5, Section 4.1.4
<i>During testing</i>		
Probe and filter temperature	Monitor and confirm proper operation	
Manometer	Check level and zero periodically	
Nozzle	Inspect for damage or contamination after each traverse	Method 5, Section 5.1
Probe/nozzle orientation	Confirm at each point	
<i>Post test checks</i>		
Sample train leak check	$\leq 0.02$ cf at highest vacuum achieved during test	Method 5, Section 4.1.4
Pitot tube leak check	No leakage	Method 2, Section 3.1
Isokinetic ratio	Calculate, must be 90-110%	Method 5, Section 6
Dry gas meter calibration check	After test series, $Y_D \pm 5\%$	Method 5, Section 5.3
Thermocouples	Same as Method 2	
Barometer	Compare w/ standard, $\pm 0.1$ " Hg	

**Table 5-6 QC Checklist and Limits for Ontario Hydro Mercury Speciation**

Quality Control Activity	Acceptance Criteria and Frequency	Reference
<i>Pre-mobilization activities</i>		
Reagent grade	ACS reagent grade	Ontario Hydro Section 8.1
Water purity	ASTM Type II, Specification D 1193	Ontario Hydro Section 8.2
Sample filters	Quartz; analyze blank for Hg before test	Ontario Hydro Section 8.4.3
Glassware cleaning	As described in Method	Ontario Hydro Section 8.10
<i>On-site pre-test activities</i>		
Determine SO <sub>2</sub> concentration	If >2500 ppm, add more HNO <sub>3</sub> -H <sub>2</sub> O <sub>2</sub> solution	Ontario Hydro Section 13.1.13
Prepare KCl solution	Prepare batch as needed	Ontario Hydro Section 8.5
Prepare HNO <sub>3</sub> -H <sub>2</sub> O <sub>2</sub> solution	Prepare batch as needed	Ontario Hydro Section 8.5
Prepare H <sub>2</sub> SO <sub>4</sub> -KMnO <sub>4</sub> solution	Prepare daily	Ontario Hydro Section 8.5
Prepare HNO <sub>3</sub> rinse solution	Prepare batch as needed; can be purchased premixed	Ontario Hydro Section 8.6
Prepare hydroxylamine solution	Prepare batch as needed	Ontario Hydro Section 8.6
<i>Sample recovery activities</i>		
Brushes and recovery materials	No metallic material allowed	Ontario Hydro Section 13.2.6
Check for KMnO <sub>4</sub> Depletion	If purple color lost in first two impingers, repeat test with more HNO <sub>3</sub> -H <sub>2</sub> O <sub>2</sub> solution	Ontario Hydro Section 13.1.13
Probe cleaning	Move probe to clean area before cleaning	Ontario Hydro Section 13.2.1
Impinger 1,2,3 recovery.	After rinsing, add permanganate until purple color remains to assure Hg retention	Ontario Hydro Section 13.2.8
Impinger 5,6,7 recovery.	If deposits remain after HNO <sub>3</sub> rinse, rinse with hydroxylamine sulfate. If purple color disappears after hydroxylamine sulfate rinse, add more permanganate until color returns	Ontario Hydro Section 13.2.10
Impinger 8	Note color of silica gel; if spent, regenerate or dispose.	Ontario Hydro Section 13.2.11
<i>Blank samples</i>		
0.1 N HNO <sub>3</sub> rinse solution	One reagent blank per batch.	Ontario Hydro Section 13.2.12
KCl solution	One reagent blank per batch.	Ontario Hydro Section 13.2.12
HNO <sub>3</sub> -H <sub>2</sub> O <sub>2</sub> solution	One reagent blank per batch.	Ontario Hydro Section 13.2.12
H <sub>2</sub> SO <sub>4</sub> -KMnO <sub>4</sub> solution	One reagent blank per batch.	Ontario Hydro Section 13.2.12
Hydroxylamine sulfate solution	One reagent blank per batch.	Ontario Hydro Section 13.2.12
Unused filters	Three from same lot.	Ontario Hydro Section 13.2.12
Field blanks	One per set of tests at each test location.	Ontario Hydro Section 13.4.1
<i>Laboratory activities</i>		
Assess reagent blank levels	Target <10% of sample value or <10x instrument detection limit. Subtract as allowed.	Ontario Hydro Section 13.4.1
Assess field blank levels	Compare to sample results. If greater than reagent blanks or greater than 30% of sample values, investigate. Subtraction of field blanks not allowed.	Ontario Hydro Section 13.4.1
Duplicate/triplicate samples	All CVAAS runs in duplicate; every tenth run in triplicate. All samples must be within 10% of each other; if not, recalibrate and reanalyze.	Ontario Hydro Section 13.4.1

## **6 DESCRIPTION OF TESTS**


Personnel from METCO Environmental arrived at the plant at 11:00 a.m. on Wednesday, October 13, 1999. After meeting with plant personnel and attending a brief safety meeting, the equipment was moved onto the Unit Number 3 Precipitator Inlet Duct and Stack. The preliminary data was collected. The equipment was secured for the night. All work was completed at 8:00 p.m.

On Thursday, October 14, work began at 6:30 a.m. The equipment was prepared for testing. The first set of tests for mercury began at 8:33 a.m. and was completed at 1:03 p.m. The samples were recovered. The second set of tests began at 2:38 p.m. The second set of tests was aborted at 5:33 p.m. due to reference method sampling equipment problems. The equipment was secured for the night. All work was completed at 8:00 p.m.

On Friday, October 15, work began at 6:30 a.m. The equipment was prepared for testing. The third set of tests for mercury began at 9:00 a.m. Testing continued until the completion of the fourth set of tests at 5:36 p.m.

The samples were recovered. The equipment was moved off of the sampling locations and loaded into the sampling van. The samples and the data were transported to METCO Environmental's laboratory in Dallas, Texas, for analysis and evaluation.

Operations at the Cinergy Corporation, Gibson generating Station, Unit Number 3  
Precipitator Inlet Duct and Stack, located in Owensville, Indiana, for the Electric Power  
Research Institute, were completed at 8:30 p.m. on Friday, October 15, 1999.

  
Billy J. Mullins, Jr. P.E.  
President